

UNITED STATES PATENT APPLICATION

OF

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AND

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FOR

**COMPOSITIONS COMPRISING AT LEAST ONE HYDROXIDE COMPOUND AND
AT LEAST ONE REDUCING AGENT, AND METHODS FOR RELAXING HAIR**

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[004] Generally, hair relaxing processes are chemical processes which may alter the aforementioned disulfide bonds between polypeptides in hair fibers and may form lanthionine residues $[S[CH_2CH(NH-)(CO-)]_2]$. Thus, the term "lanthionizing" is used when one skilled in the art refers to the relaxing or straightening of keratinous fibers by hydroxide ions.

[005] For example, hair fibers may be relaxed or straightened by disrupting the disulfide bonds of the hair fibers with an alkaline reducing agent. The chemical disruption of disulfide bonds with such an agent is generally combined with mechanical straightening of the hair, such as combing, and straightening generally occurs due to changes in the relative positions of neighboring polypeptide chains within the hair fiber. This reaction is generally terminated by rinsing and/or application of a neutralizing composition.

[006] The reaction with the alkaline agent is normally initiated by available hydroxide ions. As used herein, "available hydroxide ions" are hydroxide ions which are available for lanthionization. Not to be limited by theory, there are two reaction sequences that are predominantly used in the art to explain the disruption of the disulfide bonds in hair fibers by available hydroxide ions. Both of these reaction sequences result in lanthionine residue formation. One reaction sequence comprises at least one bimolecular nucleophilic substitution reaction wherein an available hydroxide ion directly attacks the disulfide linkage of a cystine residue. The result is the formation of lanthionine residues and HOS^- . See Zviak, C., The Science of Hair Care, pp. 185-186 (1986). The second reaction sequence comprises at least one β -elimination reaction initiated by the nucleophilic attack of an available hydroxide ion on a hydrogen atom bonded to a carbon atom that is in the β -position with respect to the disulfide bond of a cystine residue. Id. The result is the formation of a dehydroalanine residue. The dehydroalanine residue then reacts with either the thiol group of a cysteine residue or the amino group of an alanine residue to form a lanthionine residue or a lysinoalanine residue,

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respectively. These stable irreversible crosslinks in the treated hair make subsequent chemical re-linking of the polypeptides unnecessary. Thus, the only step that may be required following a straightening process using such hydroxide-containing alkaline agents is the removal of any excess alkaline solution to avoid and/or minimize damage to the hair protein or skin. If such a step is required, an acidic shampoo may be used to neutralize residual alkali and remove it from the hair and scalp.

[007] Hydroxide-containing alkaline agents also have other advantages. For example, alkaline agents, such as sodium hydroxide and guanidine hydroxide, do not have a highly objectionable odor or cause such an odor on treating the hair. Further, hydroxide-based straighteners generally have relatively fast processing times and good straightening of naturally curly or kinky hair. Additionally, the achieved straightening effect is more durable; *i.e.*, less likely to revert to a curly state after shampooing and exposure to elements than is hair straightened with some other straighteners.

[008] Despite these advantages, certain hydroxide-containing alkaline agents may have disadvantages. These disadvantages may be heightened when the hydroxide-containing alkaline agent is sodium hydroxide. Specifically, the causticity of sodium hydroxide can adversely affect the condition of the hair, for example, leaving it in a brittle state and harsh to the touch. Additionally, prolonged or unnecessary exposure of hair to such a strong alkali can weaken, break and dissolve the hair. In some instances, such a strong alkali can discolor the natural color of the hair. For example, the tone of natural brown hair may be reddened and

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natural white or grey hair may be yellowed. Further, the natural sheen of the hair may be delustered.

[009] Most frequently, commercial relaxing compositions are in the form of gels or emulsions that contain varying proportions of strong water-soluble bases, such as sodium hydroxide (NaOH), or of compositions that contain slightly-soluble metal hydroxides, such as calcium hydroxide ($\text{Ca}(\text{OH})_2$), which can be converted *in situ* to soluble bases, such as guanidine hydroxide. Traditionally, the two main hair relaxers used in the hair care industry for generating hydroxide ions are referred to as "lye" (lye = sodium hydroxide) relaxers and "no lye" relaxers.

[010] The "lye" relaxers generally comprise sodium hydroxide in a concentration ranging from 1.5% to 2.5% by weight relative to the total weight of the composition (0.38M - 0.63 M) depending on the carrier used, the condition of the hair fibers and the desired length of time for the relaxation process.

[011] While "no lye" relaxers may not contain lye, they may nonetheless rely on the soluble hydroxides of inorganic metals, such as potassium hydroxide and lithium hydroxide. Other "no lye" relaxers may use hydroxide ions obtained, for example, from a slightly-soluble source, such as $\text{Ca}(\text{OH})_2$. For example, the slightly soluble $\text{Ca}(\text{OH})_2$ may be mixed with guanidine carbonate to form guanidine hydroxide, a soluble but unstable source of hydroxide, and insoluble calcium carbonate (CaCO_3). This reaction is driven to completion by the precipitation of CaCO_3 and is, in effect, substituting one insoluble calcium salt for a slightly soluble calcium salt. Because guanidine hydroxide is unstable, the components are stored separately until the time of their use.

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More commonly, reducing agents, such as thioglycolates, sulfites, cysteines and their derivatives, are used for texturizing purposes in hair straightening or relaxing compositions. Processes comprising the application of these reducing agents generally require two steps: (1) a reducing step comprising the use of the reducing agent, and (2) a neutralizing step comprising the use of an oxidizing composition.

[014] The reaction with the reducing agent is normally initiated by thiolate ions.

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[017] The use of both reducing agents and hydroxide-based compounds has previously been disclosed. A curing method for permanent hair straightening using thioglycolic acid, dithioglycolic acid, and potassium hydroxide is known. See Ogawa, S. et al., J. Cosmet. Sci., 51, 379-399 (2000). This method comprises three steps: (1) reduction using thioglycolic acid (3% to 9%), dithioglycolic acid,

potassium hydroxide (1.05%), EDTA and monoethanolamine; (2) heat treatment, followed by (3) oxidation of the hair. Further for example, a process for imparting smoothness, body and a permanent wave pattern is also known. See U.S. Patent No. 6,058,943. This process comprises at least eleven steps such as applying an aqueous alkaline relaxant composition containing an alkaline hydroxide reducing agent to the hair (step "(a)"), applying an aqueous waving composition containing a thioglycolate reducing agent to the hair (step "(d)"), and shampooing and rinsing the hair with a neutralizing shampoo and water (step "(h)").

[018] The present invention may relax or straighten keratinous fibers without damage to the fibers but at the same time without substantial reversion to the original curly state of the hair using compositions comprising low concentrations of at least one hydroxide compound and of at least one reducing agent.

[019] Thus, the present invention, in one aspect, provides a composition for lanthionizing keratinous fibers to achieve relaxation of the keratinous fibers comprising (i) at least one hydroxide compound and (ii) at least one reducing agent chosen from thiols, sulfites, and derivatives thereof, wherein the at least one hydroxide compound and the at least one reducing agent are present in a combined amount effective to relax keratinous fibers, and with the proviso that if the at least one reducing agent is chosen from cysteine, cysteine derivatives, and thioglycolic acid, then the at least one hydroxide compound is present in an amount such that the amount of hydroxide ion is less than 1% by weight relative to the total weight of the composition. In one embodiment, the composition is heat-activated.

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[021] In another aspect of the invention, the present invention provides a method for lanthionizing keratinous fibers to achieve relaxation of the keratinous fibers comprising: (i) generating hydroxide ions in at least one solvent, wherein said step of generating comprises combining in the at least one solvent at least one hydroxide compound and at least one reducing agent chosen from thiols, sulfites, and derivatives thereof; (ii) applying a composition comprising the generated hydroxide ions to keratinous fibers for a sufficient period of time to lanthionize the keratinous fibers; and (iii) heating the keratinous fibers, wherein the at least one hydroxide compound and the at least one reducing agent are present in a combined amount effective to relax keratinous fibers, further wherein the composition is applied prior to or during heating, and with the proviso that the method does not comprise an oxidation treatment subsequent to the heating.

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hydroxide compound and at least one reducing agent chosen from thiols, sulfites, and derivatives thereof; (ii) applying a composition comprising the generated hydroxide ions to keratinous fibers for a sufficient period of time to lanthionize the keratinous fibers; and (iii) heating the keratinous fibers, wherein the at least one hydroxide compound and the at least one reducing agent are present in a combined amount effective to relax keratinous fibers, further wherein the composition is applied prior to or during the heating, and with the proviso that if the at least one reducing agent is thioglycolic acid, the at least one hydroxide compound is present in an amount less than 1% by weight relative to the total weight of the composition. The lanthionization is terminated when a desired level of relaxation of the keratinous fibers has been reached. This method may optionally further comprise other treatments, such as oxidation treatments.

[023] Further, the invention also provides for a multicomponent kit for lanthionizing keratinous fibers, wherein the kit comprises at least two components. A first component of the kit comprising at least one hydroxide compound, and a second component comprising at least one reducing agent chosen from thiols, sulfites, and derivatives thereof.

[024] Certain terms used herein are defined below:

[025] As used herein, "at least one" means one or more and thus includes individual components as well as mixtures/combinations.

[026] "Keratinous fibers" as defined herein may be human keratinous fibers, and may be chosen from, for example, hair.

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[032] As described above, the lanthionization of keratinous fibers is believed to be driven by the disruption of the disulfide bonds of cystine residues in the fibers. The

[033] Thus, the present invention provides a composition for lanthionizing keratinous fibers to achieve relaxation of the keratinous fibers comprising (i) at least one hydroxide compound and (ii) at least one reducing agent chosen from thiols, sulfites, and derivatives thereof. The at least one hydroxide compound and the at least one reducing agent are present in a combined amount effective to relax keratinous fibers. If the at least one reducing agent is chosen from cysteine, cysteine derivatives, and thioglycolic acid, then the at least one hydroxide compound is present in an amount such that the amount of hydroxide ion is less than 1% by weight relative to the total weight of the composition. In one embodiment, the composition is heat-activated. In one embodiment, the composition further comprises a cation exchange composition. In another embodiment, the composition further comprises at least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of the keratinous fibers.

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[035] The present invention also provides a method for lanthionizing keratinous fibers to achieve relaxation of the keratinous fibers comprising (i) generating hydroxide ions in at least one solvent comprising combining in the at least one solvent at least one hydroxide compound and at least one reducing agent chosen from thiols, sulfites, and derivatives thereof; (ii) applying a composition comprising the generated hydroxide ions to keratinous fibers for a sufficient period of time to relax the keratinous fibers; and (iii) heating the keratinous fibers. The at least one hydroxide compound and the at least one reducing agent are present in a combined amount effective to relax keratinous fibers. The at least one hydroxide compound may be added to a composition containing the at least one reducing agent, or vice versa. Further, the composition is applied prior to or during heating. In one embodiment, the composition is applied prior to and during heating. This method does not comprise an oxidation treatment subsequent to heating. The lanthionization is terminated when a desired level of relaxation of the keratinous

fibers has been reached. In one embodiment, the composition further comprises a cation exchange composition. In another embodiment, the composition further comprises at least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of the keratinous fibers.

[036] In yet another embodiment, the present invention provides a method for lanthionizing keratinous fibers as described above but wherein the method may optionally further comprise other treatments, such as oxidation treatments, provided that if the at least one reducing agent is thioglycolic acid, then the at least one hydroxide compound is present in an amount such that the amount of hydroxide ion is less than 1% by weight relative to the total weight of the composition.

[037] According to the present invention, the at least one hydroxide compound may be chosen from any compound comprising at least one hydroxide group which may at least partially dissociate into a counterion and a hydroxide ion in solution. Non-limiting examples of the at least one hydroxide compound include alkali metal hydroxides, alkaline earth metal hydroxides, transition metal hydroxides, lanthanide metal hydroxides, actinide metal hydroxides, Group III hydroxides, Group IV hydroxides, Group V hydroxides, Group VI hydroxides, organic hydroxides, and compounds comprising at least one hydroxide substituent which is at least partially hydrolyzable. Other non-limiting examples of the at least one hydroxide compound include sodium hydroxide, potassium hydroxide, lithium hydroxide, rubidium hydroxide, cesium hydroxide, francium hydroxide, beryllium hydroxide, magnesium hydroxide, calcium hydroxide, strontium hydroxide, barium hydroxide, cupric

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hydroxide, molybdenum hydroxide, manganese hydroxide, zinc hydroxide, cobalt hydroxide, nickel hydroxide, cadmium hydroxide, gold hydroxide, lanthanum hydroxide, cerium hydroxide, actinium hydroxide, thorium hydroxide, aluminum hydroxide, guanidine hydroxides and quaternary ammonium hydroxides. The at least one hydroxide compound can also be chosen from those formed *in situ*, such as, for example, guanidine hydroxide. As previously mentioned, guanidine hydroxide may be formed *in situ*, for example, from the reaction of calcium hydroxide and guanidine carbonate.

[038] According to the present invention, the at least one hydroxide compound may be present, for example, in an amount sufficient to effect relaxation of the keratinous fibers without damaging the fibers. According to one embodiment of the present invention, the at least one hydroxide compound is present in an amount such that the amount of hydroxide ion generally ranges from 0.05% to 3%, such as from 0.1% to 1% by weight relative to the total weight of the composition. As previously mentioned, in certain embodiments, if the at least one reducing agent is chosen from cysteine, cysteine derivatives, and thioglycolic acid, then the at least one hydroxide compound is present in an amount such that the amount of hydroxide ion is less than 1% by weight relative to the total weight of the composition.

[039] The at least one reducing agent of the present invention is chosen from thiols, sulfites, and derivatives thereof. As used herein, derivatives include salts.

Derivatives of thiols and of sulfites refers to thiols and sulfites, respectively, that are substituted with any substituent at any position of the molecule, provided that the

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reducing ability of the molecule is not substantially adversely affected. The applicability of a derivative, analog, etc. of a thiol or a sulfite may be evaluated, for example, using the procedures of Example 1. The at least one reducing agent may be chosen from thiols, sulfites and derivatives thereof, such as, for example, those listed in the International Cosmetic Ingredient Dictionary and Handbook, 8th Ed., Vol. 2 (2000) at page 1767. Non-limiting examples of suitable thiols are thioglycolates, thiolactates, thioglycerols, thiocarboxylic acids, thioesters, thioamides, alkyl mercaptans, and cysteine. In one embodiment, the at least one reducing agent is chosen from thioglycolates, and in yet another embodiment, the at least one reducing agent is chosen from ammonium thioglycolate. Non-limiting examples of suitable sulfites are hydrogen sulfite, organic sulfites such as alkyl sulfites (such as dimethyl sulfite and diethyl sulfite) and alkylene sulfites (such as glycol sulfite, 1,2-propyleneglycol sulfite, and 1,3-butyleneglycol sulfite) and inorganic sulfites (such as ammonium sulfite, magnesium hydrogen sulfite, potassium sulfite, sodium sulfite, sodium hydrogen sulfite, silver sulfite, and zinc sulfite).

[040] According to the present invention, the at least one reducing agent is generally present in an amount sufficient to complement the relaxing and/or straightening effects of the at least one hydroxide compound such that the at least one reducing agent and at least one hydroxide compound are present in a combined amount effective to relax the keratinous fibers. In one embodiment, the at least one reducing agent is present in an amount ranging from 0.1% to 5% by weight relative to the total weight of the composition. The aforementioned amounts

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[041] According to the present invention, the at least one solvent can be chosen from solvents commonly used in compositions for keratinous fibers. Non-limiting examples of the at least one solvent include water and solvents which may lower the ionic bonding forces in the solute molecules enough to cause at least partial separation of their constituent ions, such as dimethyl sulfoxide (DMSO). In one embodiment, the at least one solvent is chosen from water and DMSO. The at least one solvent can be present in an amount sufficient to ensure that, upon mixing, enough of the generated available hydroxide ions remain soluble in order to effect lanthionization of keratinous fibers.

[042] The compositions of the present invention as well as those used in the methods of the present invention may further comprise at least one suitable additive chosen from additives commonly used in hair relaxing compositions. Non-limiting examples of the at least one suitable additive include dyes, anionic surfactants, cationic surfactants, nonionic surfactants, amphoteric surfactants, fragrances, screening agents, preserving agents, proteins, vitamins, silicones, polymers such as thickening polymers, plant oils, mineral oils, synthetic oils and any other additive conventionally used in compositions for the care and/or treatment of hair.

[043] Further, these compositions may further comprise at least one cation exchange composition which may be effective in participating in the lanthionizing process. In one embodiment, the at least one cation exchange composition is

[044] These compositions may further comprise at least one complexing agent effective for dissociating the at least one hydroxide compound in an amount sufficient to effect lanthionization of keratinous fibers. The at least one complexing agent may be an agent, such as a chelating agent or a sequestering agent, that leads to a partial or full dissociation of the at least one hydroxide compound. The at least one complexing agent may chelate, sequester or otherwise tie up the counter ion of the at least one hydroxide compound, allowing more available hydroxide ions to be liberated. Of course, the at least one complexing agent may do both. In any event, the net effect of the use of at least one complexing agent in accord with the present invention is the generation of enough available hydroxide ions to effect lanthionization of keratinous fibers without relying on precipitation of a counter ion, such as Ca^{++} in the form of CaCO_3 .

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agent or itself may be a third component that is combined with one or both of the component comprising at least one hydroxide compound and the component comprising at least one complexing agent.

[046] In one embodiment, the at least one complexing agent of the present invention may be chosen from chelating agents, sequestering agents and salts of any of the foregoing. A chelating agent is a compound or ligand that can bind to a metal ion, usually through more than one ligand atom, to form a chelate. See Lewis, R.J., Hawley's Condensed Chemical Dictionary p. 240 (1997). A chelate is usually a type of coordination compound in which a central metal ion, such as Co^{2+} , Ni^{2+} , Cu^{2+} , Ca^{2+} or Zn^{2+} , is attached by coordinate links to two or more nonmetal atoms, *i.e.*, ligands, in the same molecule. Non-limiting examples of common chelating agents include ethylene-diaminetetraacetic acid (EDTA), nitrilotriacetic acid and ethyleneglycol-bis(β -amino-ethyl ether)-N,N-tetraacetic acid.

[047] Sequestering agents may be chosen from any material that prevents at least one ion from exhibiting its usual properties due to close combination with that material. Id. Certain phosphates, for example, form a coordination complex with metal ions in solution so that the usual precipitation reactions may be prevented. Id. For example, calcium soap precipitates are not produced from hard water treated with certain phosphates, or metaphosphates. Id. Other non-limiting examples of sequestering agents include hydroxy carboxylic acids, such as gluconic acid, citric acid and tartaric acid. Id.

[048] In addition, other non-limiting examples of chelating agents and sequestering agents include phosphonates, amino acids and crown ethers. In one embodiment,

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[050] Further, the at least one complexing agent may also be chosen from organic acids and salts thereof. The cations that may be used to form the salts of organic acids of the present invention may be chosen from organic cations and inorganic cations. For example, in one embodiment, the inorganic cations are chosen from potassium, sodium and lithium. In another embodiment, for example, the organic cations are chosen from cations comprising ammonium groups (such as, for example, ammonium hydroxide) and cations comprising amino groups which may form ammonium groups. In another embodiment, the organic cations are chosen from guanidine, guanidine derivatives, and amine bases. In yet another embodiment, the at least one complexing agent is chosen from mono-hydroxycarboxylic acids, dihydroxycarboxylic acids, polyhydroxycarboxylic acids, mono-aminocarboxylic acids, di-aminocarboxylic acids, poly-aminocarboxylic acids, mono-hydroxysulfonic acids, di-hydroxysulfonic acids, polyhydroxysulfonic acids, mono-hydroxyphosphonic acids, dihydroxyphosphonic acids,

polyhydroxyphosphonic acids, mono-aminophosphonic acids, diaminophosphonic acids and polyaminophosphonic acids.

[051] In a further embodiment, the at least one complexing agent is chosen from ethylene diamine tetraacetic acid (EDTA), N-(hydroxyethyl) ethylene diamine triacetic acid, aminotrimethylene phosphonic acid, diethylenetriamine-pentaacetate acid, lauroyl ethylene diamine triacetic acid, nitrilotriacetic acid, iminodisuccinic acid, tartaric acid, citric acid, N-2-hydroxyethyliminodiacetic acid and salts of any of the foregoing.

[052] In yet another embodiment, the at least one complexing agent is chosen from a salt of EDTA, such as sodium EDTA, lithium EDTA, potassium EDTA and guanidine EDTA. EDTA has a strong calcium binding constant over a wide range of pH. For example, tetrasodium EDTA generally solubilizes calcium hydroxide in aqueous media to give a clear solution. The use of at least one complexing agent, such as tetrasodium EDTA, that solubilizes the counter ion of the at least one hydroxide compound may offer the benefit of no "ashing." However, the use of one or more complexing agents that do not completely solubilize the counter ion but only form slightly-soluble or sparingly-soluble complexing agent-counter ion complexes is also within the practice of the invention.

[053] In another embodiment, the at least one complexing agent may comprise at least one "soft" entity chosen from "soft" bases and "soft" cations and at least one anion chosen from chelating anions and sequestering anions. Non-limiting examples of "soft" cations include organic cations such as guanidine. Non-limiting examples of "soft" bases include amines such as monoethanolamine,

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[056] The compositions of the present invention may be provided as one-part compositions comprising at least one hydroxide compound, at least one reducing agent, and, optionally, at least one cation exchange resin and/or at least one

[057] Other than in the operating example, or where otherwise indicated, all numbers expressing quantities of ingredients, reaction conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and in the attached claims are approximations that may vary depending upon the desired properties sought to be obtained by the present invention. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the

claims, each numerical parameter should be construed in light of the number of significant digits and ordinary rounding approaches.

[058] Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contains certain errors necessarily resulting from the standard deviation found in their respective testing measurements. The following example is intended to illustrate the invention without limiting the scope as a result. The percentages are given on a weight basis.

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[059] **Exempl** : Relaxing efficiency of naturally kinky hair treated with NaOH/thioglycolate

[060] Compositions comprising from 0.1% to 1.0% NaOH (a hydroxide compound according to the present invention) and up to 5% ammonium thioglycolate (a reducing agent according to the present invention) were prepared as shown in Table 1. A naturally kinky hair swatch was either sprayed with the thioglycolate solution or was soaked in the thioglycolate solution and then blotted dry. A hot curling iron was used to pull the hair straight for 3-12 seconds. The hair swatch was rinsed and shampooed, and then placed in a humidity chamber at 90% Relative Humidity (%RH) for 24 hours. The percent Relaxing Efficiency (%RE) is defined as

$$\%RE = (L_f / L_t) \times 100$$

where L_f = length of the relaxed hair after 24 hours at 90% RH

L_t = length of the hair at the straight configuration

The greater the relaxing efficiency (%RE), the straighter the hair after treatment.

The results are shown in Table 1.

Table 1. Relaxing Efficiency (%RE) After 24 hours under 90% relative Humidity of Hair Treated with Various Compositions

Amount of Ammonium Thioglycolate (%)	Amount of NaOH (%)				
	0.1	0.3	0.5	0.7	1
0	10%	18%	25%	36%	50%
0.1	30%	78%	80%	82%	86%
0.5	30%	86%	88%	89%	94%
1	44%	90%	93%	95%	97%
2	53%	93%	94%	96%	97%
5	54%	96%	96%	98%	98%

[061] A high relaxation efficiency after 24 hours under 90% relative humidity indicates that the hair did not display reversion. The data show that naturally curly

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hair can be effectively relaxed without substantial reversion after being treated with solutions containing low concentrations of NaOH and thioglycolate and then subjected to heat.

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